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“Step Out From the Old to the New”

IS 5316 (1998): Ammonium carbonate [CHD 1: Inorganic Chemicals]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक
अमोनियम कार्बोनेट — विशिष्टि
(पहला पुनरीक्षण)
Indian Standard
AMMONIUM CARBONATE —
SPECIFICATION
(*First Revision*)

ICS 661 : 523

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Acids, Alkalies and Halides Sectional Committee had been approved by the Chemical Division Council.

Ammonium carbonate finds extensive use in baking powders, fire extinguishers and smelling salts; for washing and defatting woollens, tanning, and separating cacao constituents as mordant in dyeing; in the manufacture of rubber articles, casein glue and casein colours; and as a reagent in analytical chemistry.

The pure ammonium carbonate $(\text{NH}_4)_2\text{CO}_3$ is formed on treating the commercial ammonium carbonate with ammonia and crystallizes. It is very soluble in water and decompose into ammonia, carbon dioxide and water on heating. It decomposes in moist air to ammonium bicarbonate $(\text{NH}_4\text{HCO}_3)$.

This standard was originally published in 1969. In this revision colorimetric method for determination of chlorides, heavy metals, phosphate, silicates and Turbidimetric method for determination of sulphur have been incorporated as an alternate method. This revision also incorporates Amendment No. 1 issued in 1981.

The composition of the committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

AMMONIUM CARBONATE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ammonium carbonate, analytical reagent and technical.

2 REFERENCES

2.1 The Indian Standards listed below contain provisions which through reference in this text, constitute provisions of this Indian Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
1070 : 1992	Water reagent grade (<i>third revision</i>)
2088 : 1983	Methods of determination of arsenic (<i>second revision</i>)

3 GRADES

3.1 The material shall have two grades, namely:

- a) Grade 1 — analytical reagent, and
- b) Grade 2 — technical.

4 REQUIREMENTS

4.1 Description

The material shall be in the form of hard, white translucent crystalline masses or cubes or powder. It shall have strong odour of ammonia, sharp taste and alkaline reaction.

4.2 The material shall also conform to the requirements specified in Table 1 when tested in accordance with the methods prescribed in Annex A.

5 PACKING AND MARKING

5.1 Packing

Grade 1 material shall be packed in bottles, and Grade 2 material shall be packed in plastic-lined drums. The containers shall be tightly closed with airtight lids.

5.2 Marking

The containers shall be marked with the following information:

- a) Name and grade of the material;

Table 1 Requirements for Ammonium Carbonate
(Clauses 4.2, 5.2.1, A-5.1.4, A-6.1.4, A-7.1.5, A-8.1.4, A-9.1.4 and A-10.1.4)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Cl No. in Annex A
		Grade 1 (3)	Grade 2 (4)	
(1)	(2)			(5)
i)	Assay (as NH_3), percent by mass, <i>Min</i>	31	30	A-2
ii)	Insoluble matter, percent by mass, <i>Max</i>	0.005	0.005	A-3
iii)	Non-volatile matter, percent by mass, <i>Max</i>	0.01	0.02	A-4
iv)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.000 2	0.002	A-5
v)	Sulphur compounds (as SO_4), percent by mass, <i>Max</i>	0.002	0.005	A-6
vi)	Phosphates (as PO_4), percent by mass, <i>Max</i>	0.001	—	A-7
vii)	Silicates (as SiO_2), percent by mass, <i>Max</i>	0.000 5	—	A-8
viii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.000 2	0.000 5	A-9
ix)	Iron (as Fe), percent by mass, <i>Max</i>	0.000 1	0.000 5	A-10
x)	Arsenic content (as As_2O_3) ppm, <i>Max</i>	2	2	A-11

- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Mass of the material, batch number and date of packing; and
- d) The words 'KEEP TIGHTLY CLOSED IN A COOL PLACE'.

5.2.1 Full analytical data for the characteristics given in Table 1 shall also be given on the label of the containers of Grade 1 material.

5.2.2 The containers may also be marked with the Standard Mark.

5.2.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions, under which the licence for the use of Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn and judged for conformity to the requirements of this specification in accordance with the procedure prescribed in Annex B.

ANNEX A

(Clause 4.2, and Table 1)

ANALYSIS OF AMMONIUM CARBONATE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF AMMONIUM CARBONATE

A-2.1 Apparatus

A-2.1.1 Glass-Stoppered Weighing Bottle

A-2.2 Reagents

A-2.2.1 Standard Hydrochloric Acid — 1 N.

A-2.2.2 Standard Sodium Hydroxide Solution — 1 N.

A-2.2.3 Methyl Orange Indicator Solution — Dissolve 0.1 g of methyl orange in 1 000 ml of water and filter.

A-2.3 Procedure

Accurately weigh a glass-stoppered weighing bottle containing 10 ml of water. Add 2.0 to 2.5 g of the sample and reweigh. Measure 50 ml of 1 N hydrochloric acid in a beaker, put the weighing bottle with the sample in it, open the stopper and wash with water. Add sufficient water to completely cover the weighing bottle and titrate the excess of acid with 1 N sodium hydroxide using methyl orange indicator.

A-2.4 Calculation

Assay (as NH_3),
percent by mass = $\frac{1.703 (50 N_1 - V N_2)}{M}$

where

V = volume in ml of sodium hydroxide consumed by excess of acid,

N_2 = normality of sodium hydroxide solution,

N_1 = normality of hydrochloric acid, and

M = mass in grams of the material taken for the test.

A-3 DETERMINATION OF INSOLUBLE MATTER

A-3.1 Procedure

Dissolve 50 g of the sample in 100 ml of water, heat to boiling and digest in a covered beaker on a steam-bath for 1 hour. Filter through a tared filtering crucible, wash thoroughly and dry at $105 \pm 2^\circ\text{C}$.

A-3.2 Calculation

Insoluble matter,
percent by mass = $\frac{100 M_1}{M_2}$

where

M_1 = mass in grams of the residue, and

M_2 = mass in grams of the material taken for the test.

A-4 DETERMINATION OF NON-VOLATILE MATTER

A-4.1 Procedure

Accurately weigh about 100 g of the sample into a tared dish, add 10 ml of water, volatilize on a steam-bath and dry for 1 hour at $105 \pm 2^\circ\text{C}$.

A-4.1.1 Reserve the residue obtained after drying for the test for heavy metals.

A-4.2 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{100 M_1}{M_2}$$

where

M_1 = mass in grams of the residue, and

M_2 = mass in grams of the material taken for the test.

A-5 TEST FOR CHLORIDES

Two methods are prescribed. Method A shall be referee method in case of dispute, and Method B the alternative method.

A-5.1 Method A (Turbidimetric Method)**A-5.1.1 Apparatus**

A-5.1.1.1 *Nessler cylinders* — 50 ml capacity.

A-5.1.2 Reagents

A-5.1.2.1 *Sodium carbonate*

A-5.1.2.2 *Concentrated nitric acid* — see IS 264.

A-5.1.2.3 *Silver nitrate solution* — approximately 4 percent (w/v).

A-5.1.2.4 *Standard chloride solution*

Dissolve 1.648 g of sodium chloride, dried at $105 \pm 2^\circ\text{C}$, in water and dilute to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of chloride (as Cl).

A-5.1.3 Procedure

Dissolve 2 g of the sample in 25 ml of hot water, add about 10 mg of sodium carbonate and evaporate to dryness on a steambath. Dissolve the residue in 20 ml of water, filter in a Nessler cylinder, and add 1 ml of nitric acid and 1 ml of silver nitrate solution. Dilute to 50 ml and mix. Carry out a control test using 0.4 ml, in case of Grade 1 material, or 4.0 ml in case of Grade 2 material, of standard chloride solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture.

A-5.1.4 The limit prescribed in Table 1 for Grade 1 and Grade 2 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than produced in the control tests.

A-5.2 Method B (Colorimetric Method)**A-5.2.1 Outline of the Method**

The residue left after elimination of volatiles of ammonium carbonate is dissolved in distilled water to which addition of mercuric thiocyanate and ferric ammonium sulphate, produces a highly coloured ferric

thiocyanate complex, the intensity of which is proportional to the chloride content. The absorbance of this colour is measured at 450 nm wavelength.

A-5.2.2 Apparatus

A-5.2.2.1 *Standard laboratory apparatus*

A-5.2.2.2 *Spectrophotometer or photocolormeter*

A-5.2.2.3 *Platinum evaporating dish*, 60 ml capacity.

A-5.2.3 Reagents

A-5.2.3.1 *Ferric ammonium sulphate solution*, 0.25 mol/l.

Dissolve 49.02 g of Ferric Ammonium sulphate dodecahydrate $[\text{Fe}(\text{NH}_4)_6(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ in 200 ml of dilute nitric acid (about 1.5 mol/l) and dilute to 500 ml with distilled water.

A-5.2.3.2 *Mercury (II) thiocyanate solution*

Saturated solution in absolute ethyl alcohol.

A-5.2.3.3 *Standard chloride solution*

Weigh accurately 0.165 0 g of previously dried (at 500°C) and cooled sodium chloride (analytical grade), dissolves in a beaker in distilled water and quantitatively transfer into a one litre volumetric flask, dilute to mark and mix well. 1 ml of this solution = 100 μg of Cl.

A-5.2.3.4 *Dilute standard chloride solution*

Transfer 50 ml of standard chloride solution (A-5.2.3.3) into a 500-ml volumetric flask, dilute with distilled water to the mark and mix well. 1 ml of this solution = 10 μg of Cl.

A-5.2.3.5 *Sulphuric acid solution*, approximately 1 mol/l.

Pour 56 ml of concentrated sulphuric acid into 944 ml of distilled water slowly, mix and cool.

A-5.2.4 Procedure**A-5.2.4.1 Calibration**

Into a series of six 50-ml volumetric flasks, transfer standard chloride solution (A-5.2.3.4) as given below:

Standard Chloride Solution, ml	Corresponding Mass of Chloride (as Cl), μg
0 (compensation)	0
2	20
4	40
6	60
8	80
10	100

Add to each, 2 ml of ferric ammonium sulphate solution (A-5.2.3.1) and 2 ml of mercury thiocyanate solution (A-5.2.3.2) in that order. Dilute to mark and mix well.

Allow to stand for 15 minutes, transfer to the cell of spectrophotometer and measure absorbance of 450 nm wavelength with compensation solution in reference cell.

A-5.2.4.2 Preparation of calibration graph

Plot chloride content in micrograms as abscissae Vs. corresponding absorbance values as ordinates.

A-5.2.5 Determination

A-5.2.5.1 Weigh suitable mass of ammonium carbonate containing 50 to 150 µg of chloride (as Cl) accurately to 1 mg and transfer into a platinum evaporating dish. Wet the sample with 20 to 30 ml of distilled water and add about 10 mg of sodium carbonate, heat it on a water bath until almost dry. Repeat wetting and heating process. Finally add a few drops of sulphuric acid solution (A-5.2.3.5) and dissolve in 20-30 ml distilled water and add about 10 mg of sodium carbonate. Transfer quantitatively into a 100-ml volumetric flask, make up to the mark with distilled water and mix well.

Filter through Whatman No. 41 filter paper and discard a small volume of initial filtrate.

Transfer suitable volume (25 to 40 ml) into a 50-ml volumetric flask, and 2 ml ferric ammonium sulphate solution (A-5.2.3.1) and 2 ml of mercury thiocyanate solution (A-5.2.3.2) in that order, dilute to mark and mix well.

Allow to stand for 15 minutes, transfer to the cell of the spectrophotometer and measure absorbance at 450 nm wavelength. Also determine chloride in blank using same volume of distilled water as that of aliquot used for test with same volume of all other reagents.

A-5.2.6 Calculations and Expression of Results

The chloride content is calculated from the formula:

$$\text{Chloride (as Cl), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10\,000}$$

where

M_1 = mass in micrograms of chloride corresponding to absorbance of test solution,

M_2 = mass in micrograms of chloride corresponding to absorbance of blank solution, and

M_0 = mass in grams of ammonium carbonate in the test portion of the aliquot used for colour development.

A-6 TEST FOR SULPHUR COMPOUNDS

Two methods are prescribed. Method A shall be referee method in case of dispute and Method B the alternative method.

A-6.1 Method A

A-6.1.1 Apparatus

A-6.1.1.1 Nessler cylinders — 50-ml capacity.

A-6.1.2 Reagents

A-6.1.2.1 Sodium carbonate

A-6.1.2.2 Concentrated hydrochloric acid — See IS 265.

A-6.1.2.3 Bromine water

Shake water with a little liquid bromine till the water itself becomes reddish brown in colour and a slight excess of liquid bromine remains at the bottom.

A-6.1.2.4 Dilute hydrochloric acid

Approximately 1 N.

A-6.1.2.5 Barium chloride solution

Approximately 10 percent (w/v).

A-6.1.2.6 Standard sulphate solution

Dissolve 0.148 g of ignited sodium sulphate (Na_2SO_4) in water and dilute to 1 000 ml. One millilitre of the solution contains 0.1 mg of sulphate (as SO_4).

A-6.1.3 Procedure

Dissolve 2 g of the sample in 20 ml of water, add about 10 mg of sodium carbonate and evaporate to dryness. Dissolve the residue in a slight excess of concentrated hydrochloric acid, add 2 ml of bromine water and again evaporate to dryness. Dissolve the residue in 4 ml of water and 1 ml of dilute hydrochloric acid. Filter in a Nessler cylinder through a small filter, wash with two 2-ml portions of water, dilute to 10 ml and add 1 ml of barium chloride solution. Dilute to 50 ml and mix. Carry out a control test in another Nessler cylinder using 0.4 ml in case of Grade 1 material, or 1.0 in case of Grade 2 material, of standard sulphate solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture.

A-6.1.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control tests.

A-6.2 Method B (Turbidimetric Method)

A-6.2.1 Outline of the Method

This describes a Turbidimetric method for the determination of sulphates and is applicable when sulphate content is less than 0.1 percent by mass.

A-6.2.2 Principle

Neutralization of the test portion (for alkaline salts) with hydrochloric acid and making it acidic with slight excess of hydrochloric acid and precipitation of sulphate as barium sulphate under well defined conditions. Measurement of turbidity using a spectrophotometer (or photocolormeter) at 470 nm.

A-6.2.3 Reagents

A-6.2.3.1 Barium chloride

A.R. Barium chloride dihydrate of uniform particle size between 0.50 and 1.25 mm, standardized by screening. It is essential that all preparations concerning the determination and standardization shall be carried out using a product of the same particle size distribution.

A-6.2.3.2 Sodium carbonate solution, approx 0.5 mol/l.

A-6.2.3.3 Hydrochloric acid solution, approx 1 mol/l.

A-6.2.3.3.1 Hydrochloric acid solution, approx 6 mol/l.

A-6.2.3.4 Standard sulphuric acid solution, 0.05 mol/l.

Prepare approximately 1 mol/l solution by pouring 56 ml of concentrated sulphuric acid into 944 ml of distilled water. Dilute 50 ml of this solution to 1 000 ml in a volumetric flask which gives approximately 0.05 mol/l solution. Standardize this against a standard sodium carbonate solution (0.05 mol/l) prepared by dissolving A.R. Sodium carbonate dried at 260-270°C. Dissolve 1.324 9 g in distilled water, transfer quantitatively into a 250-ml flask, make up to mark and mix well.

A-6.2.3.4.1 Standard sulphuric acid solution, 0.1 sulphate per ml.

Transfer 20.8 ml of 0.05 mol/l standard sulphuric acid solution (A-6.2.3.4) into a 1 litre volumetric flask, dilute to mark with distilled water and mix well. one millilitre of this solution = 0.1 mg SO₄.

A-6.2.4 Apparatus

A-6.2.4.1 Standard laboratory apparatus

A-6.2.4.2 Platinum evaporating dish, 60-ml capacity.

A-6.2.4.3 Spectrophotometer or photoelectric absorptiometer fitted with filters giving only a

negligible transmission below 450 nm and above 550 nm.

A-6.2.5 Procedure

A-6.2.5.1 Calibration

Into each of a series of nine 50-ml one-mark volumetric flasks, place the volumes of standard sulphuric acid solution (A-6.2.3.4.1) as shown in the following table:

<i>Sulphuric Acid Solution, ml</i>	<i>Corresponding Mass of SO₄, mg</i>
0	0
5	0.5
10.0	1.0
15.0	1.5
20.0	2.0
25.0	2.5
30.0	3.0
35.0	3.0
40.0	4.0

To each flask, add 2 ml of sodium carbonate solution (A-6.2.3.2) and 5 ml of hydrochloric acid solution (A-6.2.3.3.1), stir, dilute to mark and mix well.

A-6.2.5.2 Turbidimetric reaction

Transfer 25.0 ml of each of the solution except the first to separate dry 100-ml beaker each containing 0.15 g barium chloride (A-6.2.3.1). Stir by hand for 1 min at the rate of 2 rev/s, allow to stand for 15 min at 27 ± 2°C.

Stir by hand and transfer a sufficient quantity of each of the solutions to a cell of the spectrophotometer and measure the absorbance at 470 nm wavelength. Use compensation solution to adjust optical zero of spectrophotometer.

A-6.2.5.3 Preparation of calibration graph

Plot a graph with SO₄ content in mg as abscissae Vs. corresponding absorbance values as ordinates. It should be noted that the calibration curve is linear only above 0.5 mg SO₄.

A-6.2.6 Determination

A-6.2.6.1 Test portion

Weigh to the nearest 1 mg a quantity of the test sample of ammonium carbonate containing to 4 mg of SO₄ and transfer into a platinum evaporating dish (A-6.2.4.2), wet the sample with 20-30 ml of distilled water and heat on the water bath to almost dryness. Then add 5 ml of hydrochloric acid solution (A-6.2.3.3.1) and 20-30 ml of distilled water and heat again on the water bath for a few minutes.

Transfer into 50 ml one-mark volumetric flask, cool and make up to the mark with distilled water. If turbid,

filter through a Whatman No. 41 filter paper and discard a small volume of the initial filtrate.

A-6.2.6.2 Turbidimetric reaction

Transfer 25 ml filtrate (A-6.2.6.1) into a 100-ml beaker containing 0.15 g of barium chloride (A-6.2.3.1) stir by hand for 1 minute at the rate of 2 rev/s. The barium chloride should then be in complete solution.

Leave undisturbed for 15 minutes at $27 \pm 2^\circ\text{C}$.

A-6.2.6.3 Turbidity measurement

Transfer a sufficient quantity of the test solution (A-6.2.6.1) to a cell of the spectrophotometer (same size as the one used for calibration) and use to adjust optical zero of the spectrophotometer.

Stir by hand the turbidity developed solution (A-6.2.6.2), transfer it into a cell (same size as the one used for calibration) and measure absorbance at 470 nm wavelength.

NOTE — If the turbidity developed in the test portion is beyond the limits of the calibration range, dilute an aliquot filtrate (A-6.2.6.1) to 50 ml and use 25 ml of this diluted solution to develop turbidity. In this case use the diluted solution in the reference cell.

A-6.2.6.4 Blank test

At the same time, carryout a blank test following the same procedure and using the same quantities of all reagents used for determination, but by replacing the test portion by 5 ml of standard sulphuric acid solution (A-6.2.3.4.1) corresponding to 0.5 mg of SO_4 to allow operation in the linear part of the calibration curve.

A-6.2.7 Calculations and expression of results

The sulphate content is obtained by the formula

$$\text{Sulphate as } \text{SO}_4, \text{ percent by mass} = \frac{(M_1 - M_2)}{M_0} \times 10$$

where

M_1 = mass of sulphate in mg corresponding to absorbance of the test portion;

M_2 = mass of sulphate in mg corresponding to the absorbance of the blank solution after deduction of 0.5 mg of SO_4 added (that is, Graph value – 0.5)

M_0 = mass of the ammonium carbonate in g corresponding to aliquot used for turbidity development.

A-7 TEST FOR PHOSPHATES

Two methods are prescribed. Method A shall be referee method in case of dispute and Method B the Alternative Method.

A-7.1 Method A

A-7.1.1 Apparatus

A-7.1.1.1 Separating funnels — 250 ml capacity.

A-7.1.2 pH Meter — with glass and Calomel electrodes.

A-7.1.3 Reagents

A-7.1.3.1 Dilute sulphuric acid — approximately 5 N.

A-7.1.3.2 Ammonium molybdate

A-7.1.3.3 Concentrated hydrochloric acid — conforming to IS 265.

A-7.1.3.4 Dilute hydrochloric acid — approximately 5 N.

A-7.1.3.5 Ethyl ether

A-7.1.3.6 Stannous chloride solution

Dissolve 2 g of stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in concentrated hydrochloric acid and dilute to 100 ml with concentrated hydrochloric acid.

A-7.1.3.7 Standard phosphate — silicate solution

Dissolve 1.43 g of potassium dihydrogen phosphate (KH_2PO_4) and 2.365 g of sodium silicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) in water and make up to 1 000 ml. Store this solution in a waxed bottle. Dilute 1 ml of this solution to 100 ml with water immediately before use. One millilitre of this diluted solution is equivalent to 0.01 mg of Phosphate (as PO_4) and 0.005 mg of silica (as SiO_2).

A-7.1.4 Procedure

Weigh accurately 1 g of the material and dissolve in 50 ml of water in a platinum dish. Digest on a steam bath for 20 minutes. Cool, neutralize with dilute sulphuric acid to a pH of about 4, and dilute to about 75 ml. Take 1 ml of standard phosphate-silicate solution and make up to about 75 ml. Add 0.5 g of ammonium molybdate to each solution and when it is dissolved, adjust the pH to about 2 by adding dilute hydrochloric acid. Check the pH with the pH meter. Heat both the solutions to boiling, cool to room temperature, add 10 ml of concentrated hydrochloric acid to each and dilute to 100 ml with water. Transfer the solutions to separating funnels, add 35 ml of ether to each, shake vigorously and allow to separate. Draw off the aqueous phase and reserve it for determination of silica. Wash the ether phase of each by shaking with 10 ml of dilute hydrochloric acid, allow to separate, and draw off and discard this aqueous phase. Add 0.2 ml of freshly prepared stannous chloride solution to each ether extract and shake. If the ether extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

A-7.1.5 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

A-7.2 Method B (Colorimetric Method)

A-7.2.1 Outline of the Method

This method specifies a reduced molybdophosphate spectrophotometric method for the determination of soluble phosphates in presence of soluble silicates.

A-7.2.2 Principle

Silica is eliminated by precipitation and filtration. Formation of phosphomolybdic acid complex in acidic solution and reduction of yellow complex to molybdenum blue complex. Measurement of the intensity of the blue colour (which is proportional to phosphate concentration) using a spectrophotometer or photocolourimeter at 580 nm wave length.

A-7.2.3 Apparatus

A-7.2.3.1 Standard laboratory apparatus

A-7.2.3.2 Spectrophotometer or photoelectric colorimeter

A-7.2.3.3 Platinum evaporating dish, approx 60 ml capacity.

A-7.2.4 Reagents

A-7.2.4.1 Perchloric acid — 60 to 70 percent solution (m/m).

A-7.2.4.2 Ammonium molybdate solution

Dissolve 10 g of ammonium molybdate tetrahydrate $[(\text{NH}_4)_6 \text{Mo}_7 \text{O}_{24} \cdot 4\text{H}_2\text{O}]$ in 100 ml of water. Add slowly 220 ml of sulphuric acid (1:1 solution) and 70 ml distilled water, mix and allow to cool.

A-7.2.4.3 Reducing solution

Dissolve 0.25 g of 1-amino, 2-naphthol 4-sulphonic acid and 0.5 g of anhydrous, sodium sulphite (Na_2SO_3) in about 70 ml distilled water. Dissolve separately 24 g of anhydrous sodium bisulphite (NaHSO_3) in about 100 ml distilled water. Mix both solutions and dilute to 200 ml with distilled water, filter, if necessary and store in a cool dark place.

A-7.2.4.4 Standard phosphate solution

Weigh accurately 1.433 g of finely powdered and dried potassium dihydrogen phosphate analytical grade (KH_2PO_4) dissolve in 100 to 150 ml distilled water in a 250 ml beaker, transfer quantitatively to a 1 000 ml — volumetric flask, dilute to mark and mix well. Transfer 50 ml of this solution to a 500-ml volumetric flask, dilute to mark and mix well. One millilitre of this solution contains 100 μg of phosphate (as PO_4).

A-7.2.4.5 Dilute standard phosphate solution, 10 μg PO_4/l .

Transfer 50 ml of standard phosphate solution (A-7.2.4.4) into a 500-ml volumetric flask, dilute to

mark and mix well. One millilitre of this solution is equivalent to 10 μg of phosphate.

A-7.2.4.6 Hydrochloric acid solution, approximately 6 mol/l.

Add slowly 535 ml of concentrated hydrochloric acid into 465 ml of distilled water and mix.

A-7.2.5 Procedure

A-7.2.5.1 Calibration

Into a series of six 100 ml-volumetric flasks, transfer standard phosphate solution (A-7.2.4.5) as given below:

Standard Phosphate Solution, ml	Corresponding Mass of Phosphate as PO_4 , μg
0 (Compensation)	0
2	20
4	40
6	60
8	80
10	100

Add to each flask 2 ml of perchloric acid (A-7.2.4.1), 20 ml of distilled water, 10 ml of ammonium molybdate solution (A-7.2.4.2) and finally 5 ml of reducing agent (A-7.2.4.3). Make up to the mark and mix well. Place the flasks in boiling water bath for 15 minutes, cool in running water and measure absorbance using 2 cm cell in a spectrophotometer (or photocolourimeter) at 620 nm wavelength using compensation solution in the reference cell.

A-7.2.5.1.1 Preparation of calibration graph

Plot a graph with μg of PO_4 as abscissae and corresponding absorbance values as ordinates.

A-7.2.5.2 Determination

Weigh about 10 g of ammonium carbonate accurately to 1 mg (the quantity to be weighed should be such that it contains 20 to 100 μg of phosphate) and transfer into a 60-ml platinum evaporating dish, wet it with 20-30 ml distilled water and heat to dryness on a hot water bath. Repeat wetting and heating to dissociate most of the ammonium carbonate.

To the residue in the platinum dish add 20 to 30 ml distilled water and neutralize with hydrochloric acid solution and add a few drops in excess. Evaporate to nearly dryness and add 2 ml of perchloric acid (A-7.2.4.1) and again evaporate to nearly dryness. Add about 20 ml distilled water and transfer quantitatively into a 100-ml volumetric flask, cool and dilute to mark and mix well.

Filter through a Whatman No. 41 filter paper, discard a small volume of the initial filtrate and then transfer 50-ml filtrate into 100-ml volumetric flask and develop colour as described under calibration (A-7.2.5.1).

Measure absorbance at 620 nm wavelength using same size cell as used for calibration, with distilled water in the reference cell.

A-7.2.5.3 Blank test

Carryout a blank test using the same procedure and same quantities of all reagents as in the case of determination (A-7.2.5.2) except omitting the sample solution. Use distilled water instead of sample solution.

A-7.2.6 Calculations and Expression of Results

The concentration of the phosphate (as PO_4) in the material is obtained by the formula given below:

$$\text{Phosphate (as } \text{PO}_4\text{), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10\,000}$$

where

M_1 = mass in micrograms of phosphate corresponding to the absorbance of the test portion;

M_2 = mass in micrograms of phosphate corresponding to absorbance of the blank; and

M_0 = mass in grams of the ammonium carbonate content corresponding to the test portion of the aliquot solution used for colour development.

A-8 TEST FOR SILICATES

Two methods are prescribed. Method A shall be referee method. In case of dispute, Method B will be the alternative method.

A-8.1 Method A

A-8.1.1 Apparatus

A-8.1.1.1 Separating funnels — 250 ml.

A-8.1.1.2 pH metre

A-8.1.2 Reagents

A-8.1.2.1 Concentrated hydrochloric acid — conforming to IS 265.

A-8.1.2.2 n-Butanol

A-8.1.2.3 Dilute hydrochloric acid — approximately 2 N.

A-8.1.2.4 Stannous chloride solution — Dissolve 2 g stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in concentrated hydrochloric acid and dilute to 100 ml with concentrated hydrochloric acid.

A-8.1.3 Procedure

Add 10 ml of concentrated hydrochloric acid to each of the solutions reserved for the determination of silica

in A-7.1.3 and transfer to separating funnels. Add 40 ml of butanol to each, shake vigorously, and allow to separate. Draw off and discard the aqueous phase. Wash the butanol phase with three 20-ml portions of dilute hydrochloric acid, discarding the washing each time. Dilute each butanol extract to 50 ml, take a 10-ml aliquot from each and dilute to 50 ml with butanol. Add 0.5 ml of freshly prepared stannous chloride solution to each and shake. If the butanol extracts are turbid, wash with 10 ml of dilute hydrochloric acid.

A-8.1.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the blue colour produced in the test with the material is not greater than that produced in the control test.

A-8.2 Method B (Colorimetric Method)

A-8.2.1 Outline of the Method

This method specifies a reduced molybdosilicate spectrophotometric method for the determination of soluble silica. This method is applicable for material with silica content in presence of phosphate.

A-8.2.2 Principle

Depolymerization of any polymerized silica by treatment with hydrofluoric acid and boric acid. Formation of the oxidized molybdosilicate complex (yellow) under well defined conditions of acidity ($\text{pH } 1 \pm 0.05$).

Selective reduction of the complex in a strong sulphuric acid medium in the presence of oxalic acid to eliminate interference of phosphate.

Spectrophotometric measurement of the coloured complex at wavelength of maximum absorption (about 795 nm).

A-8.2.3 Reagents

A-8.2.3.1 Distilled water

A-8.2.3.2 Sulphuric acid, approx 2 mol/l.

Add 112 ml of concentrated A.R. Sulphuric acid (sp.gr.1.84) to 888 ml of distilled water slowly by constant stirring cool and mix well.

A-8.2.3.3 Sulphuric acid, approx 8 mol/l.

Add 448 ml of concentrated A.R. Sulphuric acid (sp.gr.1.84) to 552 ml of water slowly by constant stirring cool and mix well.

A-8.2.3.4 Sodium molybdate, 274 g/l solution.

Dissolve 27.4 g of sodium molybdate dihydrate ($\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$) in warm distilled water in a beaker of material free from silica of suitable capacity and after cooling dilute to 100 ml. Store, if necessary, in a bottle of material free from silica and filter, if necessary, before use.

A-8.2.3.5 Oxalic acid, 100 g/l solution.

Dissolve 10 g of oxalic acid dihydrate ($\text{COOH} \cdot \text{COOH} \cdot 2\text{H}_2\text{O}$) in 100 ml distilled water.

A-8.2.3.6 Ascorbic acid, 25 g/l solution.

Dissolve 2.5 g of ascorbic acid in water and dilute to 100 ml. Store the solution in a bottle of material free from silica and protect from light. Prepare this solution at the time of use or as an alternative.

A-8.2.3.7 Reduction solution

A-8.2.3.7.1 Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 50 ml distilled water. Then add 1.5 g of 1-amino-2-naphthol-4-sulphonic acid and dissolve by grinding.

A-8.2.3.7.2 Dissolve 90 g anhydrous metabisulphite ($\text{Na}_2\text{S}_2\text{O}_5$) in 900 ml of water.

A-8.2.3.7.3 Mix (A-8.2.3.7.1) and (A-8.2.3.7.2) and dilute to 1 000 ml. Filter if turbid and store in an opaque bottle free from SiO_2 .

A-8.2.3.8 Sodium fluoride solution, 20 g/l solution.

A-8.2.3.9 Boric acid solution, saturated solution at ambient temperature.

A-8.2.3.10 Standard silica solution, 0.2 g SiO_2 /l

Weigh to the nearest 1 mg 0.200 g SiO_2 obtained by heating pure silicic acid (H_2SiO_3) at 1 000°C until constant mass (consecutive weighings should not differ by more than 1 mg) and cooling in a desiccator into a platinum crucible of convenient capacity. Add 2 g of anhydrous sodium carbonate to the crucible, mix well preferably with a platinum spatula and carefully fuse the mixture. Add warm water directly to the crucible, heat gently until the contents are completely dissolved and transfer quantitatively to a beaker of material free from silica of suitable capacity.

Cool, dilute the solution to about 500 ml, transfer quantitatively to 1 000-ml volumetric flask, dilute to mark and mix well. Transfer the solution immediately to a bottle of material free from silica. 1 ml of this solution = 0.2 mg SiO_2 .

A-8.2.3.10.1 Standard silica solution, 2.0 μg SiO_2 /ml.

Dilute 10.0 ml of standard silica solution (A-8.2.3.10) to 1 000 ml in a 1 000-ml volumetric flask. Prepare this solution at the time of use. 1 ml of this solution = 2.0 μg SiO_2 .

A-8.2.4 Apparatus**A-8.2.4.1 Standard laboratory apparatus****A-8.2.4.2 pH meter****A-8.2.4.3 Spectrophotometer or photocalorimeter****A-8.2.4.4 Platinum crucible with lid of suitable size****A-8.2.5 Procedure****A-8.2.5.1 Preparation of the calibration graph****A-8.2.5.1.1 Preliminary test for control and correction of pH**

Place 10.0 ml of the standard silica solution (A-8.2.3.10.1) in a beaker. Add the distilled water to bring volume to 10 ml and then add 5 ml of sodium molybdate solution (A-8.2.3.4), 1 ml of sodium fluoride solution (A-8.2.3.8) and 5 ml of boric acid solution (A-8.2.3.9). Mix and measure pH with a pH meter. Adjust pH to 1.0 ± 0.05 by adding slowly using a graduated pipette or a burette drop by drop, mixing after each addition, the necessary quantity of sulphuric acid solution (A-8.2.3.2) — about 4.5 ml would be required.

Record the actual volume of sulphuric acid required and discard the solution.

A-8.2.5.1.2 Preparation of the standard colorimetric solution

Into a series of six 50-ml beakers, transfer standard silica solution as given below:

Standard Silica Solution (A-8.2.3.10.1)	Corresponding Mass of Silica
ml	μg
0*	0.0
2.0	4.0
4.0	8.0
6.0	12.0
8.0	16.0
10.0	20.0

*Compensation solution.

Add to each beaker distilled water to make up volume to 15.0 ml. Then add the volume of sulphuric acid solution (A-8.2.3.2) as calculated above (A-8.2.5.1.1) for pH correction, mix well. Then add 1 ml of sodium fluoride solution (A-8.2.3.8), mix and allow to stand for 5 min.

Colour Development

Add to each beaker 5 ml of boric acid solution (A-8.2.3.9) and 5 ml of sodium molybdate solution (A-8.2.3.4), mix and allow to stand for 10 minutes. Add 5 ml of oxalic solution acid (A-8.2.3.5) and 8 ml of sulphuric acid solution (A-8.2.3.3), allow to stand for 2 minutes. Add 2 ml of ascorbic acid solution (A-8.2.3.6) or as an alternative 2 ml of reduction solution (A-8.2.3.7.3) and allow to stand for 10 minutes. Transfer the solutions quantitatively to a series of 50 ml, volumetric flasks, dilute to mark and mix.

A-8.2.5.1.3 Measurements

Carry out the spectrophotometric measurements with spectrophotometer (or photocolorimeter) at 795 nm wavelength, after having adjusted the instrument zero absorbance with the compensation solution, use cells of 4 to 5 cm optical path length.

A-8.2.5.1.4 Preparation of the calibration graph

Plot a graph with μg of SiO_2 contained in 50 ml as abscissae and corresponding absorbance values as ordinates.

A-8.2.5.2 Determination**A-8.2.5.2.1 Test solution**

Weigh accurately correct to 1 mg an amount of ammonium carbonate (containing between 4 to 20 μg of SiO_2) and transfer into a 100-ml beaker, dissolve in minimum amount of distilled water and neutralize with 1 mol/l sulphuric acid (about 6 ml) and then add 0.5 ml acid excess, heat on a water bath to expect all CO_2 liberated and cool. Ensure that the total volume is around 15 ml. Then add the volume of sulphuric acid solution (A-8.2.3.2) used of pH correction in the preliminary test (A-8.2.5.1.1) and 1 ml of sodium fluoride solution, mix and allow to stand for 5 minutes.

Colour Development

Develop colour by the procedure given in (A-8.2.5.1.2).

A-8.2.5.2.2 Blank

Carry out a blank test following the same procedure and using the same quantities of all reagents used for the determination but replacing the test solution by the same volume of distilled water.

A-8.2.5.2.3 Spectrophotometric measurements

Carry out the spectrophotometric measurements by the procedure specified in (A-8.2.5.1.3) at 795 nm wavelength after adjusting the instrument to zero absorbance against water use the same size of the cell used for calibration.

A-8.2.5.3 Calculations and expression of results

By means of the calibration graph (A-8.2.5.1.4) determine the mass of SiO_2 content corresponding to the aliquot used for colour development.

The silica content is expressed by the relation given below:

$$\text{Silica (as SiO}_2\text{), percent by mass} = \frac{(M_1 - M_2)}{M_0 \times 10\,000}$$

where

M_1 = mass in μg of SiO_2 in the test portion of the aliquot;

M_2 = mass in μg of SiO_2 in the blank;

M_0 = mass of original material in g, the aliquot portion used for colour development.

A-9 TEST FOR HEAVY METALS

Two methods are prescribed. Method A shall be referee method in case of dispute and Method B the alternative method.

A-9.1 Method A**A-9.1.1 Apparatus****A-9.1.1.1 Nessler cylinders — 50 ml capacity.****A-9.1.2 Reagents****A-9.1.2.1 Dilute acetic acid — approximately 1 N.****A-9.1.2.2 Hydrogen sulphide solution — aqueous, saturated and freshly prepared.****A-9.1.2.3 Standard lead solution**

Dissolve 1.60 g of lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in water and dilute to 1 000 ml. Dilute 10 ml of this solution to 1 000 ml with water. One millilitre of this diluted solution contains 0.01 mg of lead (as Pb).

A-9.1.3 Procedure

Dissolve in a Nessler cylinder the residue from the determination of non-volatile matter in 5 ml of 1 N acetic acid and dilute to 50 ml. Dilute 10 ml of this solution to 40 ml. Carry out a control test in another Nessler cylinder using 4.0 ml, in case of Grade 1 material, or 10.0 ml, in case of Grade 2 material, of standard lead solution and 1 ml of 1 N acetic acid. Dilute to 40 ml. Add 10 ml of hydrogen sulphide solution to each of the Nessler cylinders.

A-9.1.4 The limit prescribed in Table 1 shall be taken as not having been exceeded, if the intensity of the colour produced in the test with the material is not greater than that produced in the control tests.

A-9.2 Method B (Colorimetric Method)**A-9.2.1 Outline of the Method**

This method gives a spectrophotometric method for the determination of lead.

A-9.2.2 Principle

Lead reacts with dithizone (diphenylthio Carbazone) to form a pink coloured complex in chloroform solution. The complex is separated by extraction with chloroform from an aqueous Ammonia-Cyanide-Sulphite solution. The absorbance of the extracted complex is measured using a spectrophotometer at 510 nm wavelength.

A-9.2.3 Apparatus**A-9.2.3.1 Standard laboratory apparatus**

A-9.2.3.2 Separating funnels of 250 ml capacity**A-9.2.3.3 Spectrophotometer or photocolormeter****A-9.2.4 Reagents****A-9.2.4.1 Standard lead solution**

Dissolve 0.799 2 g of A.R. lead nitrate $[\text{Pb}(\text{NO}_3)_2]$ in distilled water in a beaker, transfer quantitatively into a 500 ml-one mark-volumetric flask, dilute to the mark and mix well. 1 ml of this solution contains 1 µg lead.

A-9.2.4.1.1 Dilute standard lead solution

Transfer 10 ml of the above solution (A-9.2.4.1) into a 1 litre volumetric flask, dilute to mark and mix well. 1 ml of this solution contains 10 µg of lead.

A-9.2.4.2 Chloroform — Reagent grade chloroform.**A-9.2.4.3 Dithizone reagent**

50 mg/l solution in chloroform. Dissolve 5 mg of the reagent in 100 ml of chloroform.

A-9.2.4.4 Ammonia-cyanide-sulphite reagent

To 3 ml of 10 percent potassium cyanide solution (Caution — potassium cyanide solution is highly poisonous) add 35 ml of concentrated ammonia and dilute to 100 ml. Add 0.15 g of sodium sulphite and mix well.

A-9.2.4.5 Dilute hydrochloric acid solution

Approximately 1 mol/l.

A-9.2.4.6 Hydrochloric acid solution

Approximately 6 mol/l.

A-9.2.5 Procedure**A-9.2.5.1 Calibration**

Transfer standard lead solution (A-9.2.4.1.1) to a series of six 250-ml separating funnels as given below:

<i>Standard Lead Solution, ml</i>	<i>Corresponding Lead Content, µg</i>
0 (Compensation)	0
2.0	20
4.0	40
6.0	60
8.0	80
10.0	100

Add to each funnel 75 ml of ammonia-cyanide sulphite reagent solution and cautiously adjust pH to 9.5 using a pH meter (equipped with a combined electrode). This operation must be carried out slowly because, if the pH of the solution falls below 9.5 even temporarily, HCN gas may be liberated. Use of fume Cupboard during this operation is necessary. Then add 7.5 ml of dithizone reagent (A-9.2.4.3) to each separating funnel. Maintain time difference among each such that the time after addition of dithizone reagent and

final absorbance measurement is same for all. Then add 17.5 ml of chloroform (A-9.2.4.2) and shake for one minute to extract the lead complex into the chloroform layer, allow the phases to separate. Transfer the chloroform layer (lower one) through a cotton plug (placed in the stem of the separating funnel) into a 1 cm cell of the spectrophotometer. Immediately replace the lid of the cell. Measure absorbance (using chloroform extract of compensation solution, in reference cell) at 510 nm wavelength.

A-9.2.5.1.1 Calibration graph

Plot a graph with µg of lead (in 25 ml of chloroform) as abscissae and corresponding absorbance values as ordinates.

A-9.2.5.2 Determination

Weigh accurately correct to 1 mg about 10 g of ammonium carbonate sample (the quantity to be weighed, should be such that it contains 20 to 100 µg of lead) and transfer to 50-ml beaker, dissolve in about 20-25 ml distilled water and transfer quantitatively into a 250-ml separating funnel and rinse the beaker twice with 5 ml portions of distilled water. 75 ml of Ammonia-Cyanide-Sulphite reagent and then by the cautious addition of dilute hydrochloric acid solution (A-9.2.4.4), adjust pH to 9.5. Use a pH meter with combined electrode to adjust pH. This operation must be carried out slowly because if the pH of the solution falls below 9.5 even temporarily, hydrogen cyanide gas may be liberated. Use of fume cupboard during this operation is necessary. Now add 7.5 ml of dithizone reagent (A-9.2.4.2) to the separating funnel followed by 17.5 ml of chloroform. Shake for one minute to extract the lead into the chloroform layer. Allow the phase separate. Transfer the chloroform layer through a cotton plug (placed in the separating funnel stem) into a 1 cm cell of the spectrophotometer and measure the absorbance at 510 nm with chloroform in the reference cell. Read the corresponding lead content in micrograms from the calibration graph (A-9.2.5.1.1).

A-9.2.5.3 Blank test

Carry out a blank test exactly as described in (A-9.2.5.2) but excluding the test solution, use distilled water instead. Read the corresponding lead content in the blank from the calibration graph (A-9.2.5.1.1).

A-9.2.6 Calculations and expression of results

Calculated the lead content in the material from the relation given below:

$$\text{Lead content (as Pb), percent by mass} = \frac{M_1 - M_2}{M_0 \times 10\,000}$$

where

- M_1 = mass of lead in the test solution as obtained from graph in μg ;
 M_2 = mass of lead in blank as obtained from graph in μg ; and
 M_0 = mass of sample taken for analysis in grams.

A-10 TEST FOR IRON

Two methods are prescribed. Method A shall be referee method in case of dispute, and Method B the alternative method.

A-10.1 Method A (Thiocyanate Method)

A-10.1.1 Apparatus

A-10.1.1.1 *Nessler cylinders* — 50 ml capacity.

A-10.1.2 Reagents

A-10.1.2.1 *Concentrated hydrochloric acid* — conforming to IS 265.

A-10.1.2.2 *Ammonium persulphate*

A-10.1.2.3 *Ammonium thiocyanate solution* — Dissolve 30 g of ammonium thiocyanate in 100 ml of water.

A-10.1.2.4 Standard iron solution

Dissolve 0.702 g of ammonium ferrous sulphate $[(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}]$ in water containing 10 ml of dilute sulphuric acid and dilute to 1 000 ml. Dilute 10 ml of this solution to 100 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

A-10.1.3 Procedure

Dissolve 2.000 g of the sample in 5 ml of water and volatilize on a steam-bath. Dissolve the residue in 2 ml of hydrochloric acid and 20 ml of water. Filter, in a Nessler cylinder, and dilute to 40 ml. Add 30 to 50 mg of ammonium persulphate crystals and 3 ml of ammonium thiocyanate solution. Finally dilute to 50 ml and mix. Carry out a control test in another Nessler cylinder using 0.2 ml, in case of Grade 1 material, or 1.0 ml in case of Grade 2 material of standard iron solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture.

A-10.1.4 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the red colour produced in the test with the material is not greater than that produced in the control tests.

A-10.2 Method B (Bipyridyl Method)

A-10.2.1 Outline of the Method

This Indian Standard describes a 2,2'-bipyridyl spectrophotometric method for the determination of iron content.

A-10.2.2 Principle

This dissolved iron under slightly acidic medium is reduced using hydroxyl ammonium chloride and the colour is developed with 2,2' bipyridyl solution [iron (II)-2,2' bipyridyl complex]. The absorbance of the colour is measured by a spectrophotometer (or equivalent colorimeter) at 522 nm.

A-10.2.3 Apparatus

A-10.2.3.1 Spectrophotometer or an equivalent photocolorimeter suitable for measurement at 522 nm.

A-10.2.3.2 *Standard laboratory glass apparatus*

A-10.2.4 Reagents

A-10.2.4.1 Distilled water

A-10.2.4.2 *Hydrochloric acid*, approximately 1 mol/l. To 910 ml of distilled water, add 90 ml of concentrated hydrochloric acid and mix.

A-10.2.4.3 Hydroxyl-ammonium chloride solution

Dissolve 10 g of hydroxyl-ammonium chloride $(\text{NH}_2\text{OH} \cdot \text{HCl})$ in water and dilute to 100 ml.

A-10.2.4.4 *Ammonium acetate solution* : 30 g dissolved in 100 ml water.

A-10.2.4.5 2,2'-Bipyridyl solution

Dissolve 1 g of 2,2'-bipyridyl in 10 ml of hydrochloric acid solution (1 mol/l) and dilute to 100 ml.

A-10.2.4.6 Standard iron solution A

Dissolve 0.702 2 g of ferrous ammonium sulphate hexahydrate $[\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}]$ in water in a suitable beaker, add 50 ml of sulphuric acid (about 1 mol/l) and mix. Transfer quantitatively into a one-litre volumetric flask, make up to the mark and mix well. 1 ml of this solution contains 0.1 mg iron (as Fe).

A-10.2.4.7 Standard iron solution B

Dilute 100 ml of the above solution (solution A) and dilute to 1 litre in a volumetric flask, mix well. 1 ml of this solution contains 10 μg of iron (as Fe).

A-10.2.4.8 *Hydrochloric acid*, approx 6 mol/l (1:1 v/v).

A-10.2.5 Procedure

A-10.2.5.1 Calibration

Into each of a series of level 100 ml volumetric flasks, place the quantities of standard iron Solution B as given below:

<i>Standard Iron Solution B ml</i>	<i>Corresponding Iron Content as Fe µg</i>
0 (Compensation)	0
5.0 solution	50
10.0	100
15.0	150
20.0	200
25.0	250
30.0	300
35.0	350
40.0	400
45.0	450
50.0	500

Add to each volumetric flask an amount of water sufficient to dilute to approx 50 ml, then 2 ml of hydrochloric acid and 2 ml of hydroxylammonium chloride solution and after 5 minutes, add 5 ml of ammonium acetate solution and 1 ml of 2,2'-bipyridyl solution. Dilute to mark, mix well, and wait for 10 minutes.

Measure absorbance using spectrophotometer with 1 cm cell at 522 nm, using as reference the compensation solution.

A-10.2.5.1.1 Preparation of calibration graph

Prepare a calibration graph with iron as Fe in µg/100 ml as abscissa and absorbance as ordinates.

A-10.2.5.2 Determination

Weigh accurately to the nearest 1 mg, an amount of ammonium carbonate containing 100 to 1 000 µg of iron (as Fe) into a platinum evaporating dish. Dissolve in minimum amount of distilled water and heat it on a water-bath until completely dry. Again wet it with distilled water and allow to dry. This may again be repeated if necessary until all the ammonium carbonate dissociates. To the contents in the evaporating dish add a few drops of hydrochloric acid and then dilute with distilled water cool and transfer quantitatively into a 100 ml one mark volumetric flask. Make up to the mark and mix well. If the solution is

turbid filter through a Whatman No. 541 filter paper and discard 10 to 15 ml of the initial filtrate.

Transfer a suitable aliquot volume (or the filtrate if filtered) containing 50 to 500 µg of Fe into a 100-ml one-mark volumetric flask. Dilute to around 50 ml (if less), add 2 ml hydrochloric acid (A-10.2.4.2) and 2 ml of hydroxylammonium chloride (A-10.2.4.3). Mix after addition of each and after 5 minutes, add 5 ml of ammonium acetate solution (A-10.2.4.4) followed by 1 ml of 2,2'-bipyridyl solution (A-10.2.4.5). Dilute to mark, mix well and allow to stand for 10 minutes measure the absorbance using spectrophotometer or photocolormeter at 522 nm wavelength as described for calibration. Use the same cell size and as the one used for calibration and use blank test solution (A-10.2.5.3) as reference.

A-10.2.5.3 Blank

Prepare a blank test solution using same procedure as used for determination (A-10.2.5.2) but excluding the ammonium carbonate.

A-10.2.6 Calculation and Expression of Results

By means of the calibration graph (A-10.2.5.1.1) determine the mass of iron in micrograms corresponding to the absorbance value of the test solution.

Calculate the iron content as Fe in the material by the following formula:

$$\text{Iron (as Fe), percent by mass} = \frac{M_1}{M_0} \times 10\,000$$

where

M_1 = mass of iron in micrograms in the test portion; and

M_0 = mass of the material in grams corresponding to the volume used for colour development.

A-11 TEST FOR ARSENIC

Dissolve 1 000 g of the material in 10 ml of water and carry out the test for arsenic as prescribed in IS 2088 using 0.002 mg of arsenic trioxide (As₂O₃) for preparing the comparison stain.

ANNEX B

(Clause 6.1)

SAMPLING OF AMMONIUM CARBONATE

B-1 SCALE OF SAMPLING

B-1.1 Lot

In any consignment, all the containers of the same size and grade drawn from the same batch of manufacture shall be grouped together to constitute a lot.

B-1.2 For ascertaining the conformity of the material in the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number of containers to be selected for this purpose shall depend on the size of the lot and shall be in accordance with Table 2.

Table 2 Number of Containers to be Selected for Sampling
(Clause B-1.2)

No. of Containers in the Lot <i>N</i>	No. of Containers to be Selected <i>n</i>
(1)	(2)
Up to 25	2
26 to 50	3
51 " 100	4
101 " 300	5
301 and above	6

B-1.3 These containers shall be selected at random from the lot. To ensure randomness of selection, a random number table may be used. In case such a table is not available, the following procedure may be adopted:

‘Starting from any container in the lot, count them as 1,2,3,.....etc, up to *r* and so on, in one order. Every *r*th container thus counted shall be withdrawn to constitute the sample, where *r* is the integral part of N/n (*N* being the number of containers in the lot and *n* being the number of containers to be selected).’

B-2 PREPARATION OF SAMPLES

B-2.1 Draw, with an appropriate clean and dry sampling instrument, small portions of the material

from different parts of each selected container. The total quantity of the material to be drawn from each container shall not be less than 150 g.

B-2.2 The material drawn from all the selected containers according to B-2.1 shall be thoroughly mixed together to constitute a single mass. The total material so obtained shall be divided into three approximately equal parts each of which shall be called a composite sample representing the lot.

B-2.3 Each of the three composite samples obtained in B-2.2 shall be immediately transferred to appropriate sample containers which shall be sealed airtight immediately after filling and marked with necessary details for identification.

B-2.4 One of the three composite samples shall be marked for the purchaser, another for the supplier and the third kept as a referee sample.

B-2.5 The referee sample shall be kept a place and under conditions agreed to between the purchaser and the supplier. The referee sample shall be used in the case of a dispute.

B-3 CRITERIA FOR CONFORMITY

B-3.1 Tests for all the requirements of this specification shall be conducted on the composite sample.

B-3.2 The lot shall be declared as conforming to the requirements of this specification if the composite sample satisfies all the tests.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Acids, Alkalis and Halides Sectional Committee, CHD 2

<i>Chairman</i>	<i>Representing</i>
PROF P. NATARAJAN	Central Salt and Marine Chemicals Research Institute(CSIR), Bhavnagar
<i>Members</i>	
DR O. K. SRIVASTAVA (<i>Alternate</i> to Prof. P. Natarajan)	Gujarat State Fertilizers and Chemicals Ltd, Vadodara
SHRI E. L. CHRISTIE	Deepak Nitrite Ltd, Vadodara
SHRI V. V. PATEL (<i>Alternate</i>)	Punjab Alkalies and Chemicals Ltd, Nayanangal
SHRI A. G. BAJAJ	Directorate General of Supplies and Disposals, New Delhi
SHRI D. P. GUPTA (<i>Alternate</i>)	Standard Alkalies, Chemical Division, Mumbai
SHRI S. C. TANDON	Grasim Industries Ltd, Nagda
SHRI H. S. GAS (<i>Alternate</i>)	Indian Soap and Toiletries Makers' Association, Mumbai
SHRI P. NAIR	Shriram Foods and Fertilizers Industries, New Delhi
SHRI N. K. KAUSHAL (<i>Alternate</i>)	Indian Dyestuff Industries Ltd, Mumbai
SHRI V. K. KAPOOR	Gujarat Alkalies and Chemicals Ltd, Vadodara
SHRI A.S. KULKARNI (<i>Alternate</i>)	National Test House, Calcutta
SHRI P. R. MARATHE	Office of the Salt Commissioner, Jaipur
SHRI S. C. JAIN (<i>Alternate</i>)	Bengal Chemicals and Pharmaceuticals Ltd, Calcutta
SHRI V. P. MENON	Punjab National Fertilizers and Chemicals Ltd, Chandigarh
SHRI A. K. MEHRA	Shriram Fertilizers and Chemicals, Kota
SHRI RAJIV MARWAH (<i>Alternate</i>)	Department of Industrial Development, Ministry of Industries, New Delhi
DR S. R. DESHMUKH	Ballarpur Industries Ltd, Karwar
SHRI H. D. DESAI (<i>Alternate</i>)	Indian Paper Mills Association, Calcutta
SHRI J. B. SHARMA	Tata Chemicals Ltd, Mumbai
SHRI H. G. NAIK (<i>Alternate</i>)	Alkali Manufacturers' Association of India, Mumbai
DR B. B. PAUL	Saurashtra Chemicals, Porbandar
SHRI YOGESH CHANDRA (<i>Alternate</i>)	E.I.D. Parry (India) Ltd, Chennai
SHRI R. PRAKASH	Ministry of Defence (DGQA), Kanpur
SHRI R. MOHAN (<i>Alternate</i>)	Southern Petrochemicals Industries Corporation Ltd, Chennai
SHRI P. ROY	Hindustan Levers Ltd, Mumbai
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(Continued on page 16)

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DR R. S. RAJAGOPALAN,
Director (Chem)

Representing

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The Dharamsi Morarji Chemicals Co Ltd, Mumbai

Director General, BIS (*Ex-officio Member*)

Member-Secretary

SHRI R. P. SINGH
Deputy Director (Chem), BIS

Composition of Alkalies and Allied Products Subcommittee, CHD 2 : 2

Convener

SHRI J. B. SHARMA

Members

SHRI H. G. NAIK (*Alternate to*
SHRI J. B. Sharma)
SHRI SOHAN SINGH
SHRI J. S. TALWAR (*Alternate*)
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SHRI H. U. DOSHI (*Alternate*)

Gujarat alkalis and Chemicals Ltd, Vadodara

Ballarpur Industries Ltd, New Delhi

Central Salt and Marine Chemicals Research Institute (CSIR),
Bhavnagar

Chemplast Sanmar Ltd, Mettur Dam

Deepak Nitrite Ltd, Vadodara

Department of Industrial Development, Ministry of Industry, New Delhi

Indian Paper Mills Association, Calcutta

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